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SPECIAL METALS CORPORATION

New Hartford, New York

"Evolution of a Commercial 400 Ksi
Grade Maraging Steel"

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By

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PRINTED ABSTRACT

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In the relatively short span of about 10 years maraging steels were conceived and developed into highly respected engineering alloys. This type of steel has not reached its full strength potential and this presentation details the evolution of the latest production maraging steel; a 400 Ksi yield grade. This steel is a member of a new compositional group which has demonstrated yield strength capability in excess of 500 Ksi in the laboratory. The 400 Ksi yield grade was chosen as the first steel of this group for production development.

The composition and phases present in this ultra high strength steel are discussed. Mechanical properties and structures of wrought bar made from 15 pound laboratory vacuum induction melts are compared with those of bar stock made from a 16-inch vacuum arc remelted ingot weighing more than 2000 pounds. The influence of thermal treatments upon properties and structure is discussed in relation to the fabrication of finished parts made from the production melted 400 Ksi yield maraging steel.

INTRODUCTION: Start

Yield strengths of some alloy steels have reached the 400 to 450 Ksi level through application of special working techniques such as ausforming. The 18%-nickel maraging steels have attained yield strengths approaching 350 Ksi by direct heat treatment of conventionally wrought products. In the fall of 1966 Mihalisin and Bieber* of the International Nickel Company revealed results of an exploratory program designed to determine the extent to which iron-nickel maraging systems could be advanced toward higher strength levels. The approach was via composition changes to produce strength by heat treatment alone. Several compositions with strengths in excess of 350 Ksi were delineated and two of the best are compared with the 18%-nickel maraging steels in Figure 1. Nickel FeB, cobalt FeB and molybdenum FeB are still the principal alloying elements but that percentage of each is varied significantly. As indicated, nickel content is reduced whereas cobalt and molybdenum contents are increased. FeB Titanium is still used in minor amounts but aluminum is not added. Small experimental heats of the 400 and 500 types exhibit high strength, but the 500 type has very marginal ductility. Ductility FeB of the 400 type approximates that of alloys in the 18%-nickel series which currently attains only about 350 Ksi yield strength.

Special Metals became interested in 400 Ksi yield strength alloy because it appeared to offer a strength advantage over existing maraging steels while maintaining a reasonable degree of ductility. Fifteen pound experimental heats were made in a small vacuum induction melting furnace and rolled on a pilot plant mill into 5/8" square barstock. Properties essentially duplicated the International Nickel experimental results with the exception of tensile ductility which was only about half of that reported in their original investigation (Figure 2). Communications with International Nickel Company revealed that the lower ductility values are not abnormal for the 400 Ksi composition. The heat treatment used in this program was a direct aging of the hot worked barstock for four hours at 900°F followed by an air cool. This treatment conforms to that used in early work by International Nickel in the prior investigation.

PRODUCTION SCALE-UP:

Several inquiries were received regarding the 400 Ksi composition and the remaining laboratory material was distributed to others for evaluation. Continued early interest prompted Special Metals Corporation to consider scale-up of the 400 Ksi composition to a production size heat.

A nominal 2000 pound heat size was selected as being large enough to demonstrate production capability. A schematic of the production processing cycle is compared with laboratory processing of the smaller heats in Figure 3. Whereas the experimental heats were only single vacuum melted and directly rolled from ingot to barstock, vacuum induction melting plus vacuum arc remelting were selected for the large heat in order to assure a reproducible, homogeneous product. Selected

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* "Progress Toward Attaining Theoretical Strength with Iron-Nickel Maraging Steels", J. R. Mihalisin and C. C. Bieber, Journal of Metals, September 1966.

virgin raw materials were vacuum induction melted and cast into a 14" diameter electrode mold. After cropping and conditioning, the electrode was remelted in a consumable electrode furnace to a 16" diameter ingot. This ingot was press cogged to an 8" square billet and hot sheared to yield two equal pieces. Each piece was forged to 6-1/2" square billet which is a convenient reroll size. Specific practices were based on experience obtained during conversion of the 15 pound ingots and suggestions from the International Nickel Company. The 6-1/2" square stock was conditioned and rolled to 1/2" round bar in the production bar mill to supply stock for mechanical property evaluation.

Through the vacuum melting process, chemistry control of the large heat is very accurate and very low residual element and gas level are attainable. Figure 4 shows the chemistry analysis of the heat as compared with the aim chemistry. Results listed for the major elements are from a dip sample taken just prior to pouring the vacuum induction melt. These results were duplicated with good accuracy on samples from the 6-1/2" square billet and 1/2" diameter barstock. Results also confirmed that good homogeneity was achieved and that processing had not resulted in detectable loss of any element. Gas analyses run on the half-inch barstock indicated the very low levels shown, even after complete processing.

PROPERTY EVALUATION:

For initial property evaluation of the large heat, the hot rolled bar was direct aged so that results could be compared with previous data from the laboratory heats. Results of this evaluation which are tabulated in Figure 5 are compared with laboratory heat data. Properties listed for the 2000 pound heat represent the range of properties obtained from testing samples taken from several hot worked 1/2" diameter bars. Some strength scatter exists but the data closely reproduce results from the smaller laboratory heats and give the first indication of a successful scale-up to a meaningful production quantity.

Since early interest in this alloy was for fastener applications, notch-to-smooth tensile characteristics were evaluated early in the program. Specimen blanks from the 1/2" barstock were aged and then machined into notch tensile bars with varying notch severity. Figure 6 shows the results of testing. At a K_t of 2, the notch-to-smooth ratio is slightly greater than one. The ratio decreases to about 0.83 at K_t of 2.5 and 0.73 at K_t of 3.5 although nothing beyond K_t of 5 was tested. The notch behavior appears to level out beyond this point. It appears from these data that notch characteristics will be satisfactory for many fastener applications but some special design attention may be required.

In order to assess the high temperature capabilities and determine maximum useful temperature, a series of elevated temperature tensile tests were conducted. These were performed both as short time tests (30 minute hold at temperature prior to test), and as prolonged exposure tests

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(200 hour soak at temperature) to assess the stability of the material. FeB
Results of this study are shown in Figure 7. Short time tensile strength decreases gradually with increasing temperature to about 800^oF and then declines rapidly. FeB Even with the decrease in strength, the grade exhibits FeB yield strength above 300,000 psi at 800^oF. With a 200 hour soak at temperature, the strength decreases more slowly with increasing temperature and the exposed samples are actually 20-40 Ksi stronger than the unexposed samples in the 700^oF - 800^oF region. It is important to observe that this strength advantage is accompanied by only a slight decrease in ductility. The room temperature strength after 200 hour exposures at various temperatures is shown in Figure 8. Room temperature tensile strength increases significantly with a 200 hour exposure at 700^oF or 800^oF. Ductility does not deteriorate appreciably as a result of prolonged elevated temperature exposure. Beyond 800^oF, strength drops off drastically and it appears 800^oF will be the upper service temperature limit for the alloy.

HEAT TREAT STUDIES:

Two factors observed in the data presented led to consideration of heat treat studies to improve upon the direct aging of hot worked material. These are:

1. Some scatter in tensile data in both smooth and notch tensile tests suggests consideration of an annealing or stress relieving treatment to reduce variations in property response believed caused by localized stress differences induced during hot working and cold straightening.

2. The tensile data from 200 hour exposure studies suggests beneficial strengthening and perhaps better ductility from aging treatments below 900^oF.

A brief heat treatment study was conducted in which samples were solution heat treated FeB for one hour at 50^oF intervals FeB between 1800^oF and 2000^oF and air cooled, followed by a four hour aging treatment at 900^oF. Tensile tests of these samples indicated that all heat treatments weakened the material to various degrees and solution heat treatments above 1900^oF promoted grain growth FeB and considerable loss of strength. A treatment of 1800^oF for one hour was chosen for additional evaluation because it offered the least amount of strength deterioration and no grain growth, while hopefully providing a high degree of stress relief. Figure 9 shows the relationship of room temperature properties for the alloy in various heat treatment conditions. The material is quite ductile in the as-rolled condition. Annealing FeB for one hour at 1800^oF reduces the strength and hardness but does not appear to affect ductility. Aging the as-rolled structure produces the full hardness and strength. Aging the 1800^oF solution annealed structure gives strengths approximately 15,000 psi lower but ductility appears about the same. Hardness is slightly lower.

FeB To study the effect of lower aging temperatures, a series of hardness curves using 800^oF, 900^oF and 1000^oF aging temperatures after the one hour solution heat treatment at 1800^oF were developed and are

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presented in Figure 10. These curves verify that the lower 800°F aging temperature has the potential of developing higher hardness and consequently strength than a 900°F aging temperature. Maximum hardness was developed in ten hours at 800°F. The R_c 60 hardness achieved at 800°F suggests that the 15 Ksi strength loss resulting from the 1800°F/1 hour solution heat treatment can be recovered with a lower aging temperature. This is relatively recent data and mechanical tests have not yet been conducted to verify the indicated strength improvement as a result of lower aging temperature.

METALLOGRAPHY:

Preliminary metallographic work including conventional and electron microscopy has not been very rewarding. Attempts to identify the hardening phase have been unsuccessful. However, from a consideration of the chemistry, the primary hardening phase is believed to be a Mu phase of the Fe_7Mo_6 type. Figures 11 and 12 respectively show typical unetched and etched structures of hot rolled bar aged for four hours at 900°F and air cooled. The larger particles shown in Figure 11 are most likely MC carbides which range in size from 0.4 to 0.06 microns.

FeB

In the background a finer, spheroidal particle dispersion can be seen which may be the hardening phase. In the etched structure more of these particles can be observed.

APPLICATIONS:

FeB Much more work needs to be done in developing and understanding the 400 Ksi maraging steel. However, the alloy to date has exhibited many favorable and attractive characteristics that portend to its potential usefulness. These characteristics outlined in Figure 13 are detailed below.

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1. The alloy has tensile strength in excess of materials currently available, combined with usable ductility.

2. The alloy has elevated temperature capability to 800°F with good stability on the basis of 200 hour exposures.

3. Hardnesses in excess of R_c 60 are obtainable at room temperature. Young's modulus for this alloy is about 30 million Psi at room temperature and remains at 30 million Psi to 600°F. In contrast, the 18%-nickel maraging steels have a modulus of only 26-28 million Psi at room temperature which decreases rapidly at elevated temperatures.

4. From observations during production of the large heat, workability of this material appears to be in the same category as that of the 18%-nickel maraging steels. Also, heat treatment is equally uncomplicated. A short age after hot working will produce full strength. For reproducibility, the alloy appears receptive to a simple annealing treatment prior to aging. No quenching is necessary; the alloy throughhardens during air cooling.

5. Perhaps the most important characteristic is a demonstrated scale-up capability of the alloy with reproduction of chemistry and properties.

Consideration of the alloy's favorable characteristics suggests several potential applications which are listed in Figure 14. Hardness and elevated temperature strength suggest bearing applications. High strength and good stiffness suggest tooling fixtures. Formability and strength with reasonable notch strength suggest fasteners. Perhaps the alloy may find application in certain high strength areas where 18%-nickel series maraging steels are used such as rocket motor cases, retainer rings and pressure vessels.

CLOSING REMARKS:

In conclusion, it appears that many materials are inhibited in their development and application because they never become more than a laboratory curiosity. The main purpose in this work is to take a unique new material with appealing properties out of the laboratory and introduce it to production. The successful attainment of this goal eliminates the major problem of large scale material availability which restricts design and application engineers from evaluating an attractive new material.

	18 NICKEL SERIES	400 TYPE	500 TYPE
Nickel	18	13	8
Cobalt	7-9	16	18
Molybdenum	3-5	10	14
Titanium	0.2-0.8	0.2	0.2
Aluminum	0.1	---	---
Iron	Balance	Balance	Balance
UTS	210000-370000	400000	495000
0.2 YS	200000-350000	390000	486000
Elong., %	10-30	8	1
R.A., %	30-80	42	3
Hardness, R_c	40-59	62	67

FIGURE 1

Chemistry and property comparison of two newer maraging steels with the more common 18 nickel maraging system.

	AIM COMPOSITION	X-2755	X-2756
Carbon	0.03 max.	.025	.030
Nickel	13	12.9	12.9
Molybdenum	10	9.9	10.0
Cobalt	15	15.0	15.0
Titanium	0.2	0.19	0.20
Iron	Balance	Balance	Balance
UTS		409000	408000
0.2 YS		408000	397000
Elong., %		4.4	4.1
R.A., %		29.2	20.4
Hardness, R_c		61	61

FIGURE 2

Chemistry and properties of 15 pound VIM heats of a 400 Ksi experimental maraging steel.

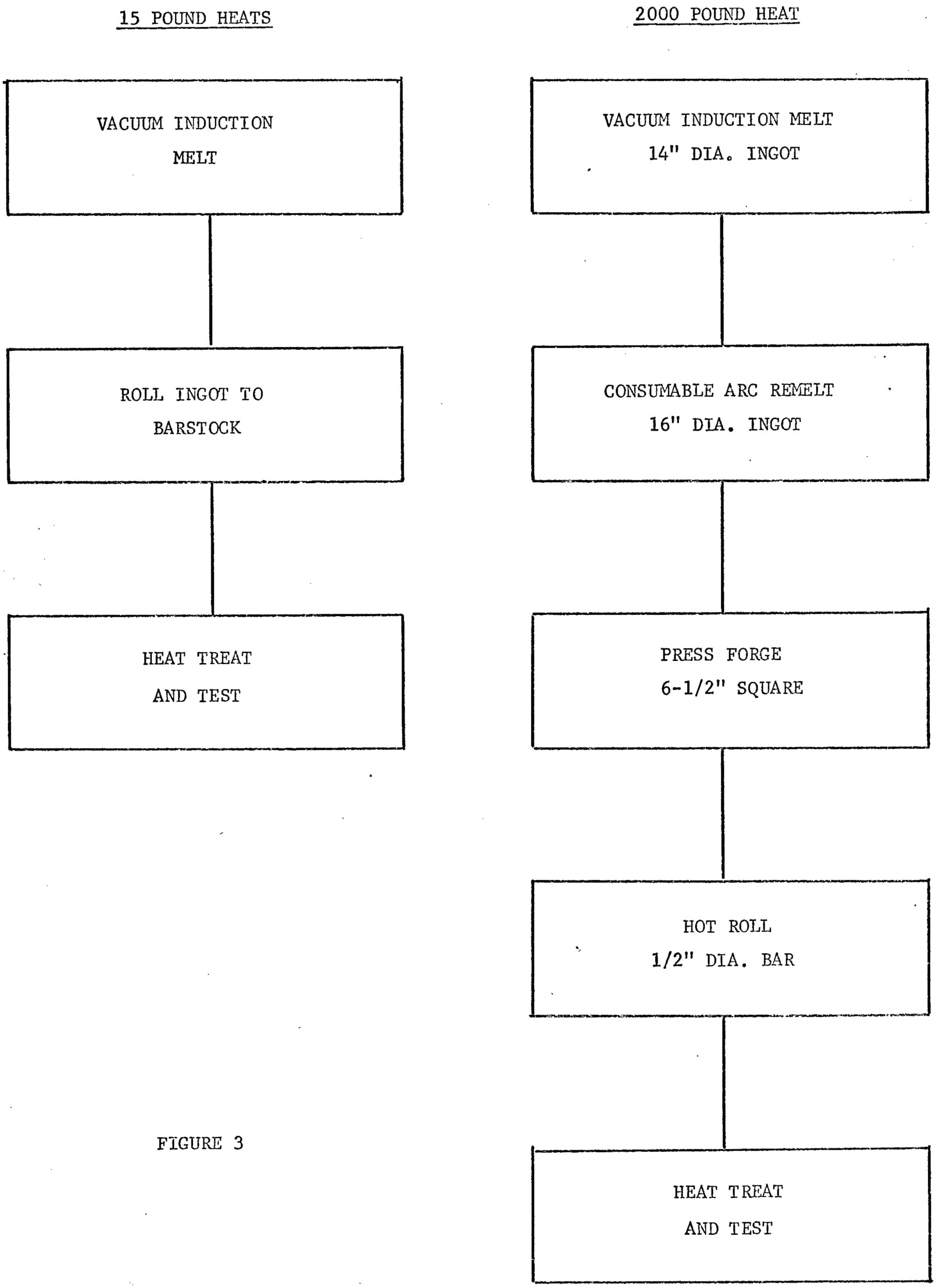


FIGURE 3

Processing sequence for 15 pound and 2000 pound heats of a 400 Ksi experimental maraging steel.

ELEMENT	GRADE AIM	2000 POUND HEAT 6-5599
CARBON	0.03	0.026
NICKEL	13.0	13.0
COBALT	15.0	15.0
MOLYBDENUM	10.0	10.1
TITANIUM	0.2	0.2
IRON	BALANCE	BALANCE
MANGANESE	<0.1	0.05
SILICON	<0.1	NIL
SULFUR	<0.01	0.002
PHOSPHOROUS	<0.01	0.001
NITROGEN	---	6 PPM
OXYGEN	---	4 PPM
HYDROGEN	---	0.4 PPM

FIGURE 4

Aim vs. actual chemistry for a 2000 lb. VIC heat of experimental maraging steel.

HEAT	UTS	0.2 YS	ELONG., %	R.A., %	HARDNESS, R_c
SPECIAL METALS 15 LB. HEATS	409000	408000	4.4	29.2	61
	408000	397000	4.1	20.4	61
SPECIAL METALS 2000 LB. HEAT	398000	391000	4.8	28.8	61
	415000	407000	4.3	21.3	61
	413500	404500	4.6	26.2	61

FIGURE 5

Comparison of properties between 15 pound VIM heats and a 2000 pound VIC heat of a 400 Ksi experimental maraging steel. Properties from hot rolled barstock aged 4 hours at 900°F, air cooled.

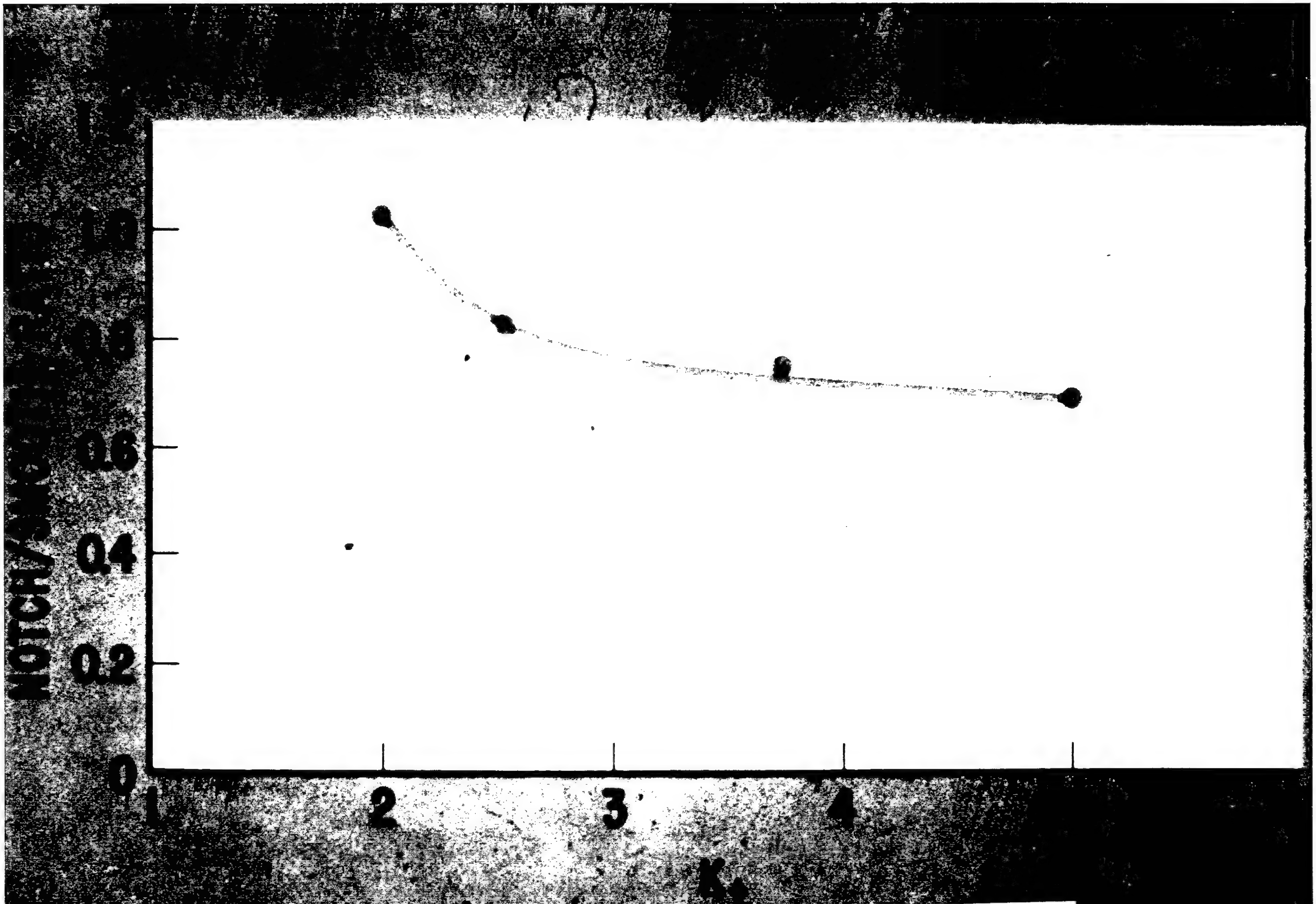


Fig. 6
Notch Toughness of a 400 Ksi experimental maraging steel. Heat 6-5599; 1/2" diameter barstock aged 4 hours at 900°F, air cooled. (Note: reproduced from 2" x 2" color slide)

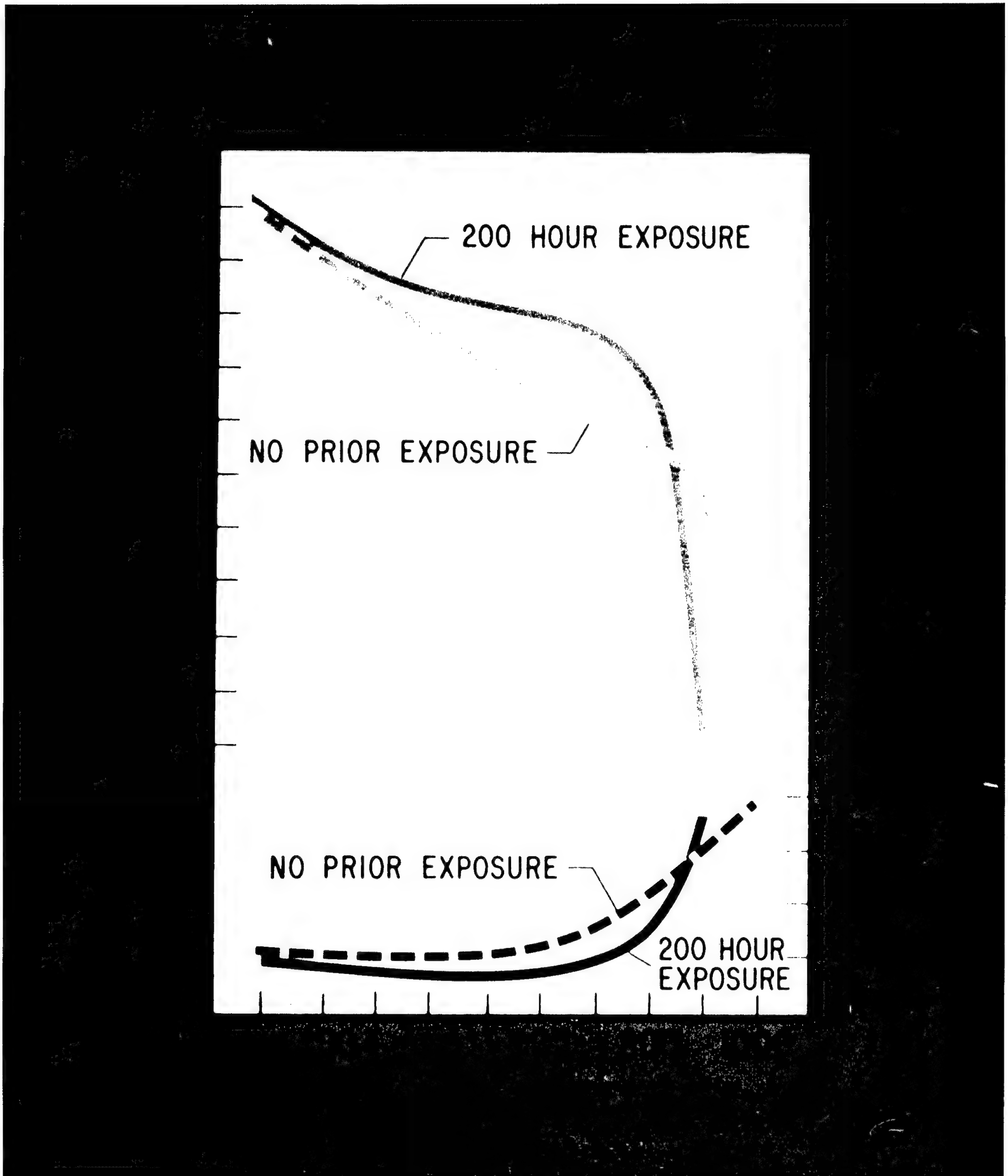


Fig. 7
Elevated tensile properties of an experimental 400 Ksi maraging steel before and after 200 hour exposure at test temperature. Heat 6-5599; 1/2" diameter barstock aged 4 hours at 900°F, air cooled. (Note: reproduced from 2" x 2" color slide)

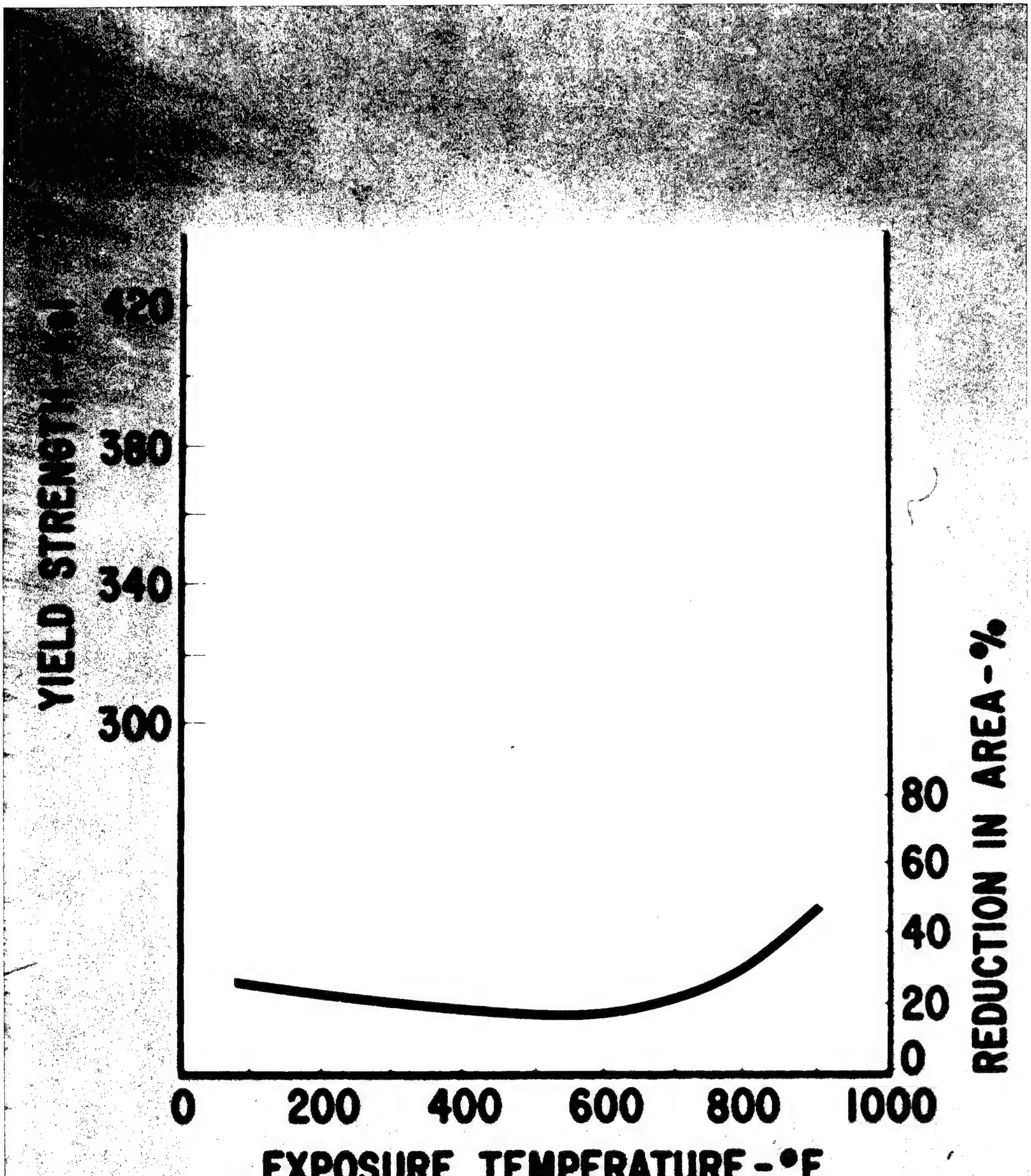


Fig. 8
Effect of 200 hour exposure at various elevated temperatures on room temperature properties of a 400 Ksi experimental maraging steel. Heat 6-5599; 1/2" diameter barstock aged 4 hours at 900°F, air cooled. (Note: reproduced from 2" x 2" color slide)

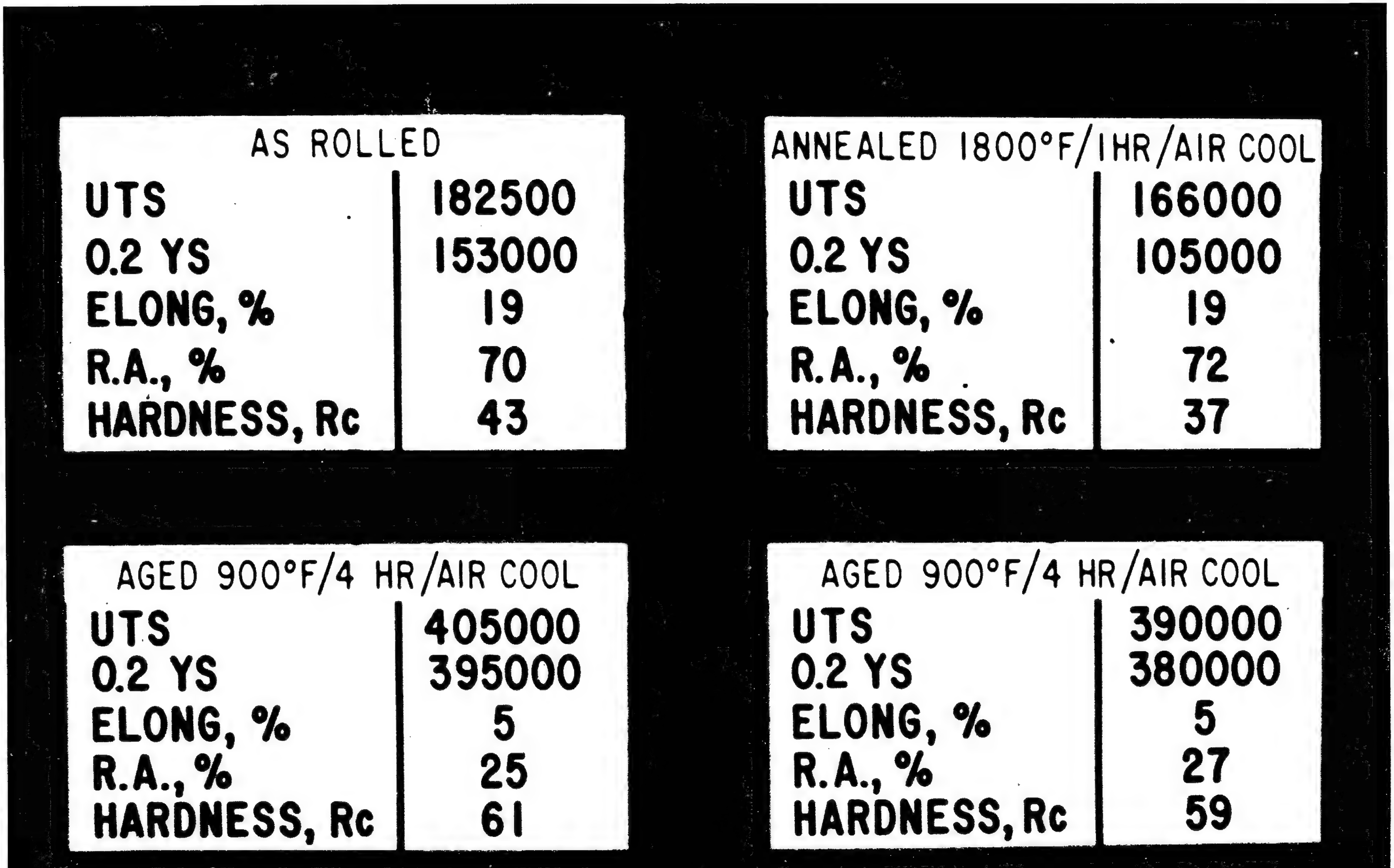


Fig. 9

Properties of an experimental 400 Ksi maraging steel in various heat treated conditions. Heat 6-5599; 1/2" diameter barstock. (Note: reproduced from 2" x 2" color slide)

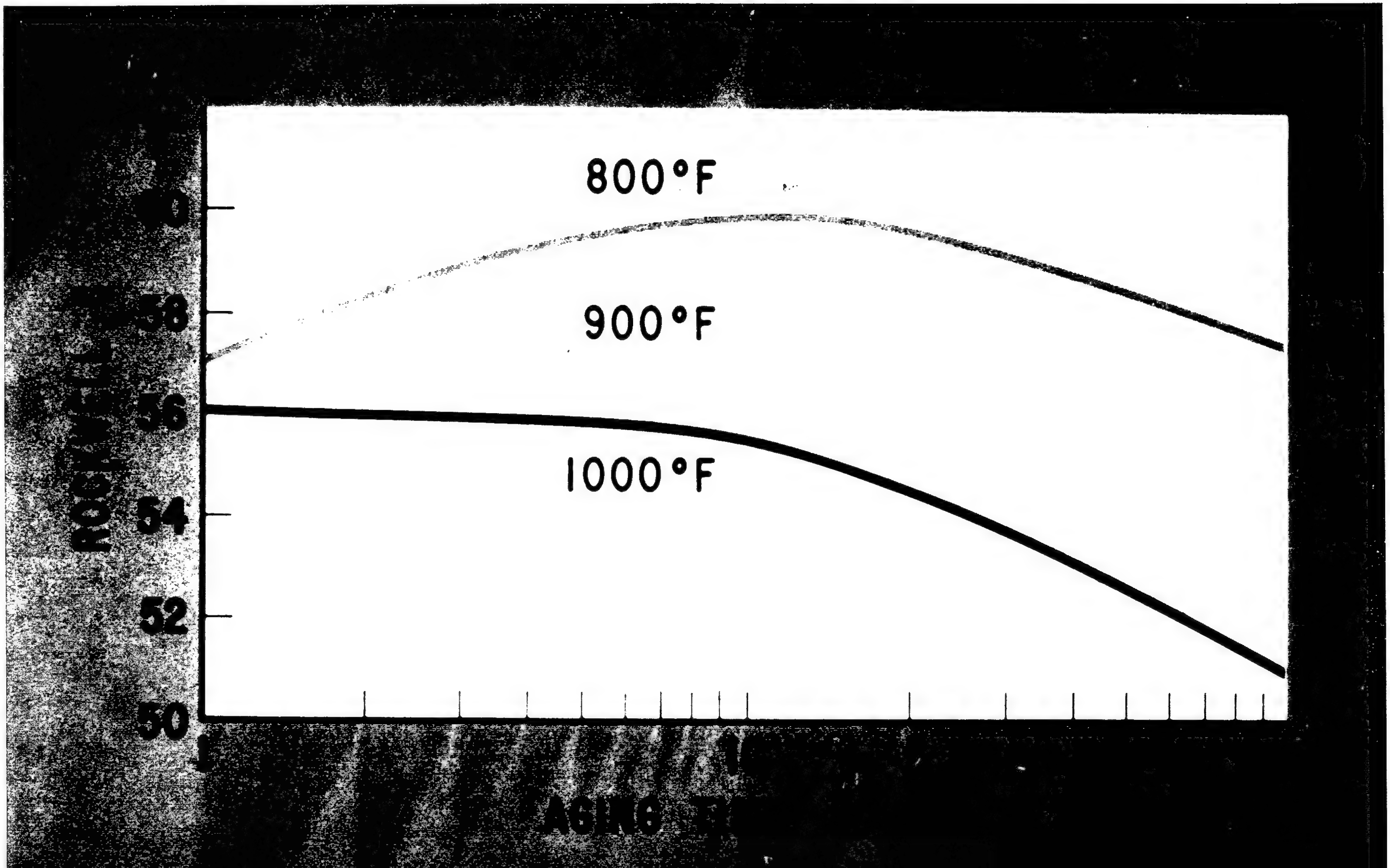
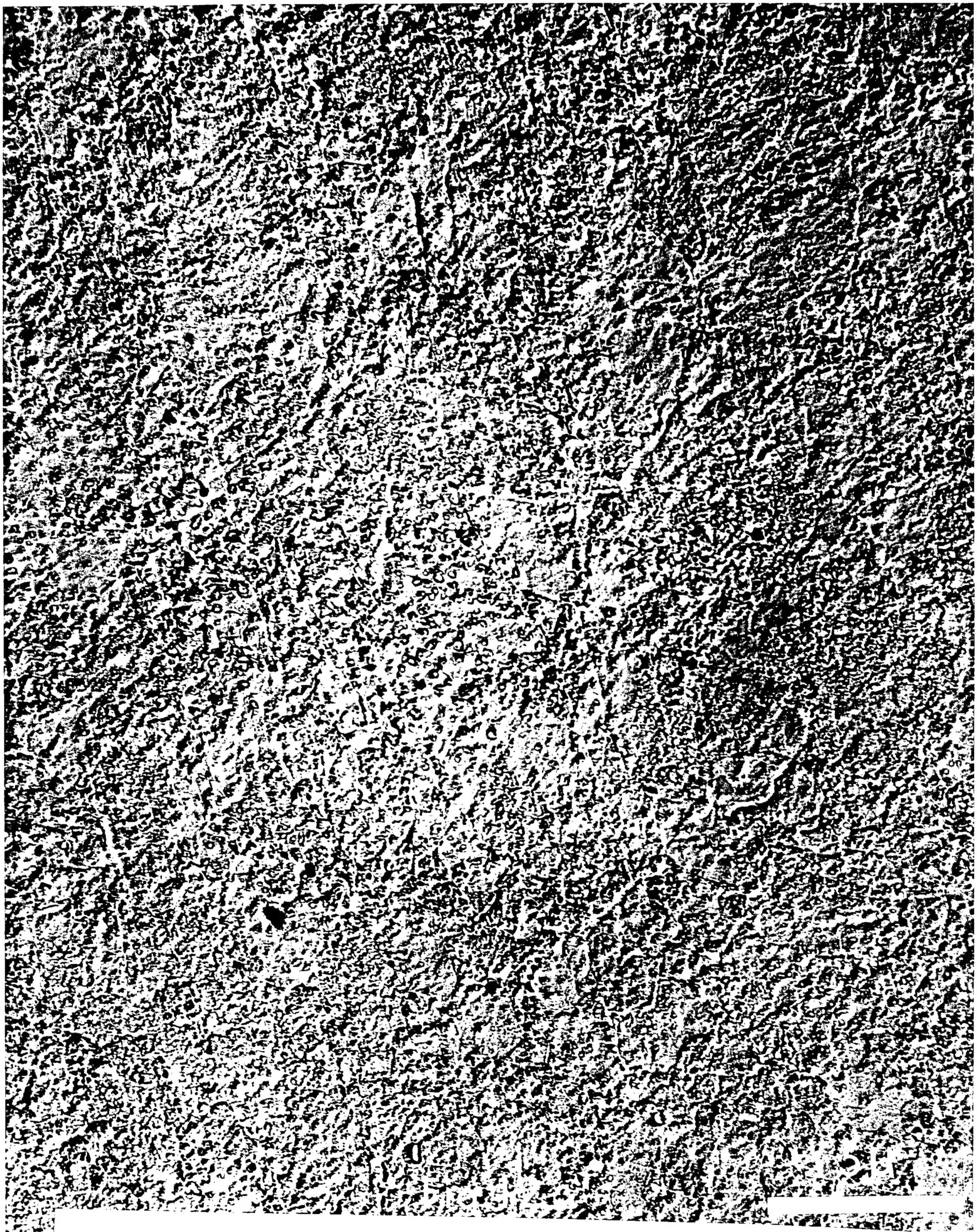


Fig. 10

Effect of aging time and temperature on hardness of an experimental 400 Ksi maraging steel. Heat 6-5599; 1/2" diameter barstock heat treated for 1 hour at 1800°F, air cooled. (Note: reproduced from 2" x 2" color slide)

Unetched microstructure of an experimental 400 Ksi maraging steel. Heat 6-5599; 1/2" diameter barstock aged 4 hours at 900°F, air cooled.



Etched microstructure of an experimental 400 Ksi maraging steel. Heat 6-5599; 1/2" diameter barstock aged 4 hours at 900°F, air cooled.

FAVORABLE CHARACTERISTICS

1. HIGH STRENGTH WITH USABLE DUCTILITY
2. ELEVATED TEMPERATURE STRENGTH AND STABILITY
3. HIGH HARDNESS AND STIFFNESS
4. EASE OF FABRICATION AND HEAT TREATMENT
5. DEMONSTRATED PRODUCTION SCALE-UP

FIGURE 13

Favorable characteristics of a 400 Ksi experimental maraging steel based on preliminary evaluation of a 2000 pound VIC heat 6-5599.

POTENTIAL APPLICATIONS

FOR A 400000 PSI LEVEL

MARAGING STEEL

BEARINGS

FIXTURES & TOOLING

PRESSURE VESSELS

ROCKET MOTOR CASES

RETAINER RINGS

FASTENERS

OTHER?

FIGURE 14